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Article

Miniaturized Optoelectronic Measurement System for Decentralized Agrifood Quality Control

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Abstract: Food safety and quality are the first steps in the food chain. This work reports a low-cost and easy-to-use optoelectronic measurement system for improving feed chain quality and safety, based on near-infrared spectrometry (NIRS) technology. This is a significant challenge for dairy farm technicians and producers who need rapid and reliable knowledge of forage quality on their farms. In most cases, instrumentation suitable for these specifications is expensive and difficult to operate. The core of the measurement system is Texas Instruments´ NIRscan Nano Evaluation Module (EVM) spectrometer. This module has a large sensing area and high resolution suitable for forage samples. To evaluate the feasibility of the prototype to analyze agrifood samples, different ways of presenting the sample, intact or ground, were tested. The final objective of the research is not just to check the efficiency of the proposed system. It is also to determine the measurement system characteristics and how to improve them.

Keywords: agrifood quality control; digital micromirror device (DMD); forage; near-infrared spectroscopy (NIRS)

1. Introduction

Animal feed's nutritional value is essential for quality and safe feed consumption and animal welfare. In addition to this fact and due to the great variability of the raw materials used to feed animals, it is necessary to develop strategies focused on tight controls of animal feed products. These should be together with the research and development of new, simple, economical, and robust methods for monitoring quality and safety parameters.

Any edible part of the plant that can be harvested or fed to animals, other than separate grains, is known as forage. It is one of the main feed products in animal husbandry and must therefore be subject to safety and quality controls. Among the most important quality parameters of forages, the following three can be highlighted. Fiber content is mainly provided by the fodder cell wall and represents its carbohydrate. Mineral content (ash) gives information about possible contamination with soil. and supplies micronutrients to the diet, it also provides information on the quality of the forage. Finally, the protein content, the importance of which lies in its influence on animal production.

Near Infrared spectroscopy (NIRS) techniques have always been valued and used for forage quality analysis [1–6] due to the speed of analysis, the simplicity of sampling, the non-invasive nature of the technique [7–9] and the possibility to be implemented in the production line [10]. In addition to the advantages of this technique, the possibility of developing miniaturized NIR systems, easy to use and specialized in quality control of raw forages used in animal feed, makes it possible to increase quality control (sampling). The use of easy to use portable NIRS instrumentation minimize time losses, because nonspecialized personnel are required to analyse samples on-site and a real-time

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response is achieved as soon as analysis is carried out. These mentioned characteristics are some of those required in precision farming.

Taking into account all these considerations, this work evaluates the feasibility of a low-cost (<$1,000) optoelectronic measurement system prototype to analyze forage samples. The core of the measurement system is a NIR spectrometer based on the Texas Instruments NIRscan Nano Evaluation Module (EVM). This module has a large sensing area and high resolution to analyse forage samples. To evaluate the feasibility of the prototype, different ways of presenting the sample, intact (raw) or ground, were studied.

This equipment has already been tested for its use in liquid sample analysis [11], with a specific cuvette for liquid samples. For this purpose, alfalfa samples were analysed. Alfalfa is one of the main forages used for animal feeding, due to its high biomass production, protein, and fiber contents. In summary, this work contributes:

- To confirm the efficiency of the proposed measurement system.
- To find out what are the qualities of the equipment.
- To look for aspects to improve and implement in the future.

The remainder of this paper is organized as follows: Section 2 introduces the main features of the measurement system developed. Section 3 focuses on methods for analysing dairy farm forage quality. Section 4 reports on experimental and discussion. Section 5 contains conclusions.

2. Materials

2.1. NIRscan Nano Spectrometer

The core of the optoelectronic measurement system is the Texas Instruments NIRscan Nano evaluation module (EVM) [12]. The block diagram of this module for reflectance measurements is shown in Figure 1. A slit collects and concentrate diffuse reflections by illuminating samples at an angle which eliminates specular reflections. Light passing through the slit is collimated, low-pass filtered, and then dispersed into constituent wavelengths by a reflective grating. Each wavelength is represented by a separate image created by the focusing lens of the digital micromirror device (DMD).

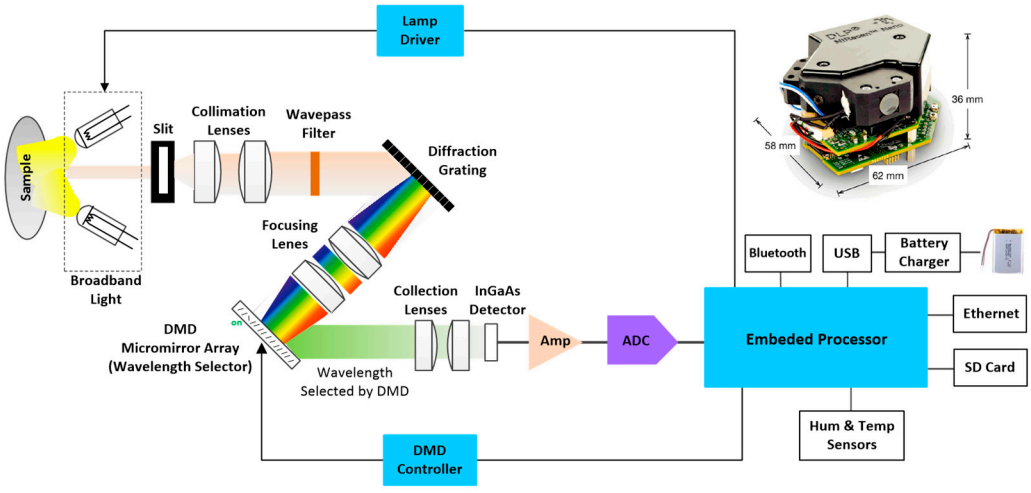

Figure 1. Block diagram and image (right upper corner) of the Texas Instruments DLP NIRscan Nano EVM.

DMD is controlled by the embedded processor to turn on and off only certain mirrors at a certain time. The width of the DMD columns selected as "on" determines the amount of light directed to the photodetector, as well as the resolution of the system. DMD columns selected as "off" divert unselected wavelengths away from the photodetector's optical path to prevent interference. By doing this, they are able to achieve high signal-to-noise ratios (SNRs).

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An array detector can't take advantage of adaptive scanning techniques, which can be performed with this type of architecture. Light energy is collected and concentrated by the collection lenses onto the single-point InGaAs photodetector. Analog-to-digital converters (ADCs) convert photodetector signals to digital values through transimpedance amplifiers.

2.2 Measurement System

The instrument sample window can be enlarged if raw samples with larger particle sizes are analyzed. This will produce reproducible spectra. However, this is difficult when measuring them directly since the sample window is very small (10 mm x 10 mm). This problem was overcome by attaching a semicircular sample holder to the spectrometer, as shown in Figure 2a. To ensure homogenous results in the sample, 10 measurements are made at each holder position. Figure 2b shows the microcontroller (ESP32) attached to the spectrometer module that drives the servomotor (MG90S). This servo motor is powered by a 3.7 to DC-DC converter (Pololu U1V0F5). By pressing the start button, the servo motor rotates and stops at different positions to make measurements. Following 180º, the holder returns to its original position and waits for another measurement to take place. By pressing the load button, the servo motor rotates 90º to an intermediate position, making loading and unloading easier. In order to minimize power consumption, the microcontroller goes into sleep mode when neither of these two actions is occurring.

(**a**) (**b**)

Figure 2. Measurement system with (**a**) whole sample, (**b**) Microcontroller board attached over the spectrometer module.

3. Methods

3.1. Forage Samples

In this study, a calibration set of 57 samples of hay or dehydrated alfalfa collected in north of Spain were involved. All samples were scanned in their initial state, then processed with a domestic spice mill (cheap and easy to use) and re-scanned in their ground form. Nutritive value parameters were achieved by analyzing all samples using reference procedures. Neutral Detergent Fiber (NDF), through Van Soest analysis [13], Mineral content (MC) through gravimetric assay and Crude Protein (CP) through Kjeldahl analysis. In Table 1 are summarizes the statistics for nutritive parameters of all the samples involved in this study, the range and variability of each analyzed parameter.

Table 1. Statistics for Nutritive Value of Alfalfa Samples (N = 57).

Parameter (%)	Mean	Min	Max	SD
NDF	40.70	29.24	61.01	5.50
MC	10.64	8.47	13.71	0.91
CP	14.48	7.19	17.01	1.70

NDF: Neutral detergent fiber; MC: Mineral content; CP: Crude protein.

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3.2. Spectral Acquisition

Spectral acquisition requires a scan configuration. Texas Instruments provides two different scan configurations from the factory, "Column" and "Hadamard". Column scanning selects wavelengths one at a time. With Hadamard scanning, several wavelengths are multiplexed at a time, and wavelengths are decoded individually. Hadamard scanning collects light and offers higher SNR, but it depends heavily on the spectrum being measured and the measurement system [14]. Column analysis is more effective for analyzing forage samples because it is more accurate than other methods of analysis due to reproducibility.

A spectrum plot and scan configuration parameters are shown in Figure 3. Before scan collection, from 1 to 5 sections can be configured depending on the type of method (Column or Hadamard), the spectral range (starting wavelength and ending wavelength), the digital resolution (wavelength points captured within the defined spectral range) the exposure time (between 0.635 and 60.960 milliseconds) and the number of scans per sample (in this work, 10 scans at 10 different points of the sample).

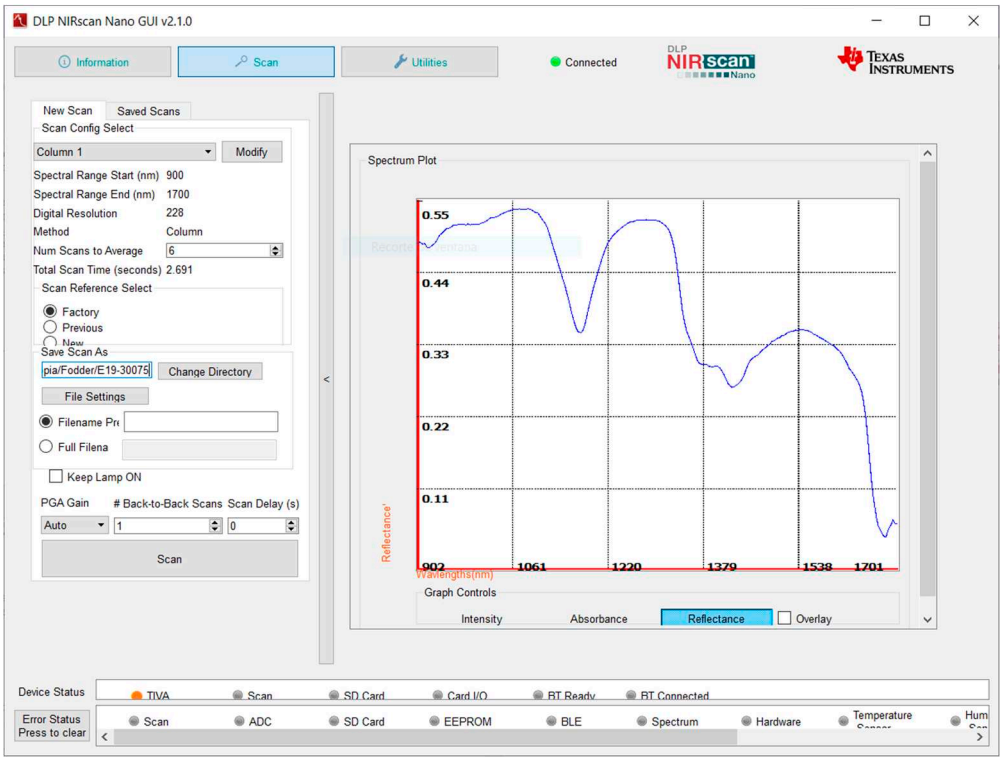

Figure 3. NIRscan Nano GUI scan screen (reflectance signal).

In this work, all samples were scanned in reflectance mode, using the measurement system of Figure 2. Each spectrum is an average of 10 spectra in a wavelength range between 901-1700nm, with a non-linear path wavelength ranged between 2.9 to 3.9 nm. The precision of collected spectrum for each sample or signal reproducibility, was evaluated for raw and ground alfalfa. The statistic, root mean square error (RMS) was calculated [15,16]. Using Equation 1 it is possible to calculate RMS for each sample spectrum. Lower RMS values are related to reproducible and repeatable spectra.

$$RMS = 10^6 \times \sqrt{\frac{\sum D^2}{n}}; D = y_a - y_b \quad (1)$$

y_a = log 1/R to λ for the average spectrum resulting from averaging a number of scans and R is Reflectance.

y_b = log 1/R to λ for the average spectrum resulting from averaging b number of scans

n = number of spectral data

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3.3. Spectra Data Processing

To establish a calibration model to quantify alfalfa nutritive parameters, different chemometric strategies were assayed. The software Unscrambler X was employed to find the linear correlation between NIRS spectra and nutritive parameters. This software takes NIRS spectra and transforms them into a matrix with X and Y variables, defined as wavelength and reflectance. To detect potential spectral outliers, a principal component analysis (PCA) was performed on the calibration set, before constructing regression models using partial least squares (PLS) [17]. An optimal number of PLS factors is determined by Unscrambler X software package, based on the minimum residual variance and 20 segments. Different spectra math pretreatment strategies were used for NDF, MC and CP quantification. These approaches were performed both using the full range of equipment from 900 to 1700 nm and a reduced one from 900 to 1600 nm.

To establish a successful model, a combination of pretreatments, including scatter correction with Standard Normal Variate (SNV), 1st and 2nd Savitzky Golay derivatives (SG) were tested in this study. Six possible pre-treatments were developed for three parameters, studying two possible wavelength ranges, for both raw and milled samples. A total of 72 mathematical pre-treatments were assayed.

To evaluate and select the most suitable chemometric model, the following statistics were calculated: coefficient of determination for calibration (R^2, see Equation 2) and standard error of calibration (SEC, see Equation 3) [18].

$$R^2 = 1 - \frac{\sum_{i=1}^{n}(y_i - \hat{y}_i)^2}{\sum_{i=1}^{n}(y_i - \bar{y})^2} \quad (2)$$

y_i are the reference values obtained in the laboratory, $\hat{y}_i$ is the prediction of the model, and n represents the number of samples used in the calibration set.

$$SEC = \sqrt{\frac{1}{n}\sum_{i=1}^{M}(y_i - \hat{y}_i)^2} \quad (3)$$

This parameter provides an average of the typical uncertainty for future sample prediction based on $\hat{y}_i$ and y_i values for sample i^{th}.

For each parameter evaluated, the best mathematical pre-treatment was selected for raw and ground NIR sample analysis. The criteria for the selection of these pre-treatments were based on the lowest values of calibration standard error (SEC) as well as the values closest to 1 for calibration determination coefficients (R^2) [19].

4. Results and Discussion

To understand a NIRS procedure, the characterization of the spectra data obtained with the NIRscan nano prototype is essential. Figure 4 shows raw and SNV plus 1st Savitzky Golay derivative spectra of alfalfa samples involved in the calibration procedure. Within the NIR wavelength range of the NIRscan nano prototype, we can identify some characteristic bands of forages [7]. According to the bibliography, those bands observed at 1166 nm are related to the protein content of the samples [19] and those observed around 1350 nm are related to the fibre content [20]. And around 1400 nm there is a band that can give information about moisture content because at that wavelength OH bond overtone vibrations are observed [7]. Figures 4 and 5 have been highlighted with a rectangle all the cited wavelengths and the referenced respective parameters. Hence, in Figure 4B, which is an extended area between 1650 and 1700 nm of Figure 4A we can observe that some of the collected spectra show a noisy signal at the end of the collected spectrum. This noisy wavelength range, as shown in Figure 4C, can be minimized after applying scattering correction (SNV) and other math pretreatments, such as derivatives.

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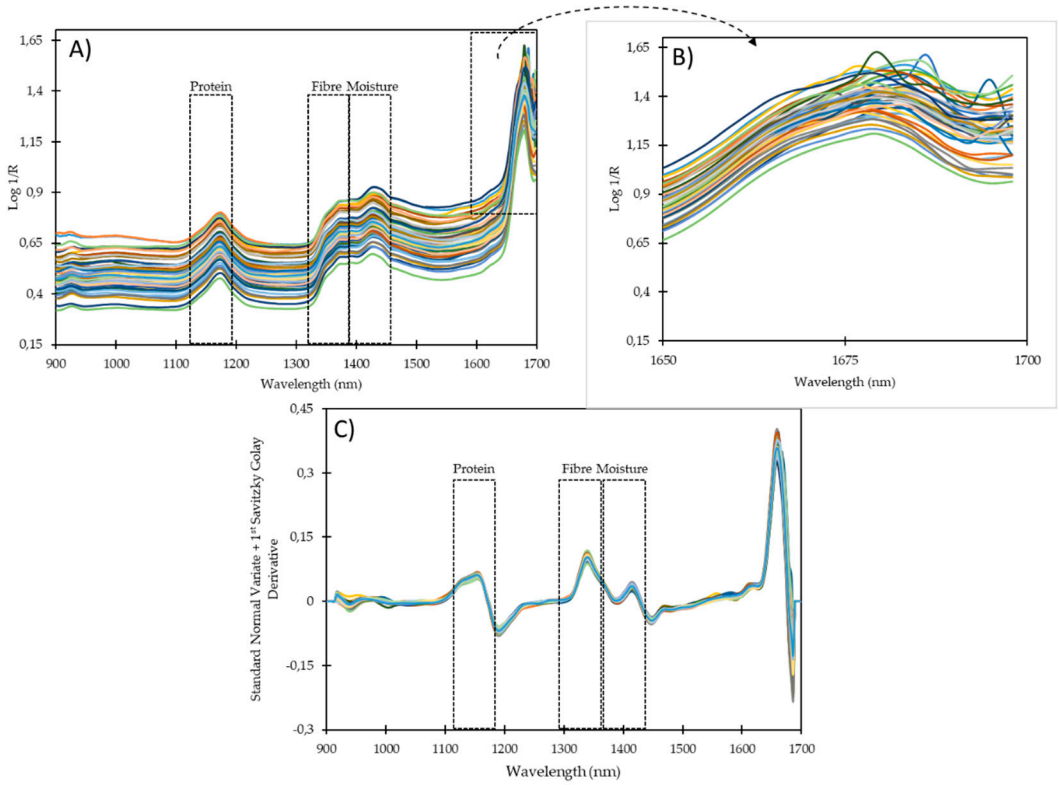

Figure 4. Spectra of the whole samples: (**A**) raw spectra; (**B**) Extended area from 1650 to 1700nm; (**C**) Standard Normal Variate +1st Savitzky Golay derivative pretreatment.

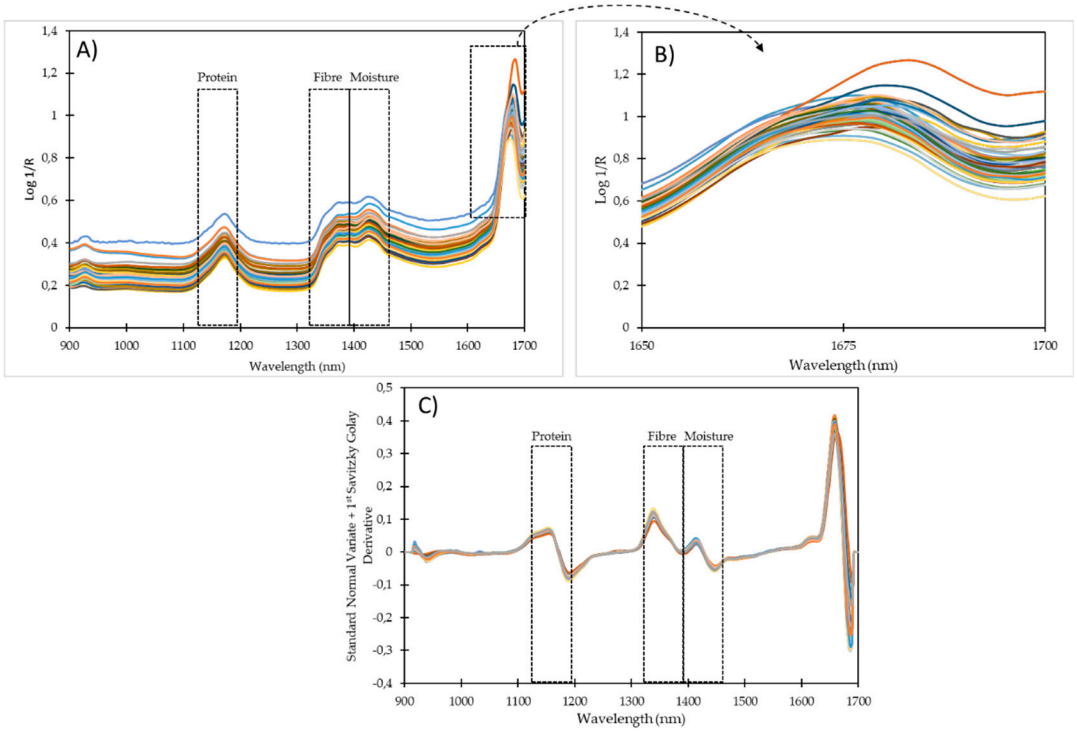

Figure 5. Spectra of the ground samples: (**A**) raw spectra; (**B**) Extended area from 1650 to 1700nm; (**C**) Standard Normal Variate + 1st Savitzky Golay derivative pretreatment.

To evaluate the effect of the sample pretreatment on the spectra data set, ground samples were scanned with NIRscan nano. Figure 5A shows the spectra data set. As can be seen, no differences in the representative bands are observed. Moreover, the extended wavelength range (1650-1700 nm, see Figures 4B and 5B) shows that after milling the sample pretreatment the noisy wavelength range

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disappears. These data confirm that spectra quality depends on the sampling procedure (raw or ground). This distorted wavelength range is due to the huge and non-homogeneous particle size of alfalfa samples. It is worthy to mention, that after applying math pretreatments no differences were observed in the collected spectra of alfalfa samples (see Figures 4C and 5C).

As observed in Figures 4 and 5, between 1600 and 1700 nm the absorbance increases, and the SNR is lower than in other ranges. It is because the intensity measured at the detector is proportional to the number of DMD mirrors positioned to reflect the incident illumination towards it. As the number of pixels changes, the measured intensity is affected as well, resulting in an increase in noise levels.

Once the spectra were evaluated, the precision of the subsampling procedure for each scanned sample (raw and ground alfalfa) was evaluated [21]. Five samples were randomly selected from the 57 analysed. The RMS value (Equation 1) was calculated for both intact and ground samples with the two ranges proposed (901–1700 nm and 901–1600 nm). Results are shown in Table 2.

Table 2. Root mean square error (RMS) values for paired subsamples of the same scanned sample.

	901–1700 nm		901–1600 nm	
Sample	**Raw**	**Ground**	**Raw**	**Ground**
1	60529	27209	43283	14560
2	58378	23853	33386	10886
3	54190	20057	34085	7029
4	54854	30655	36262	17678
5	48472	33325	34239	21939

Once the values have been calculated, two clear trends can be observed. As expected, the RMS values obtained for intact samples (raw) are higher than those obtained for ground ones. These results could be due to raw alfalfa heterogeneity. When comparing the entire range or suppressing the last 100 nm of the spectrum, we also observed a significant difference. Table 2 shows that RMS values ranged from 900 to 1600 nm are lower than the full ranging values. These results highlight the influence of the sampling procedure on spectra data precision.

After characterizing the spectral signal, the next step was to develop a calibration model. To attempt calibration, it is necessary to build a data matrix including nutritive values (NDF, MC and CP) and spectra data. After that, prior to carrying out calibrations, as mentioned in the Material and Methods section, different mathematical pre-treatments were applied for the three parameters on raw and ground samples, both for the full range and the reduced range. Partial Least Square Regression is used to establish the correlation between spectra and assayed parameters.

Table 3 summarizes NIRS models' calibration statistics to quantify NDF. As can be seen in Table 3, R^2 values are higher and SEC values are lower in chemometric models developed with the reduced wavelength range than in those developed using the full one. Related to the variability of the results depending on the math pretreatment, it is important to remark that SNV plus the second Savizky Golay derivative reached the best calibration statistics for raw and ground samples. Previous authors [22] after evaluating different commercial portable NIRS instruments to analyze ground forages, for NDF obtained R^2 values ranging between 0.95-0.71 depending on the employed instrumentation. Regarding SEC values, their results were between 2.85-1.21. Being not possible to obtain SEC values lower than 1 because the standard error of the laboratory (SEL) for this parameter is higher than 1.3 [22].

Table 3. Calibration statistics of NIR multivariate models for Neutral Detergent Fiber quantification.

	Raw alfalfa			
Wavelength Range:	**901-1600 nm**		**901-1700 nm**	
Math pretreatment	**R^2**	**SEC**	**R^2**	**SEC**
1 4 4 SG	0.898	1.554	0.883	1.670
2 4 4 SG	0.784	2.184	0.786	2.289

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SNV 1 4 4SG	0.911	1.392	0.791	2.187
1 4 4 SG SNV	0.840	1.910	0.145	1.398
SNV 2 4 4SG	0.955	1.066	0.514	3.155
2 4 4 SG SNV	0.726	2.558	0.540	3.238
Ground Alfalfa				
Wavelength Range:	**901-1600 nm**		**901-1700 nm**	
Math pretreatment	**R^2**	**SEC**	**R^2**	**SEC**
1 4 4 SG	0.756	2.749	0.598	2.830
2 4 4 SG	0.842	2.258	0.623	3.371
SNV 1 4 4SG	0.761	2.694	0.043	5.383
1 4 4 SG SNV	0.796	2.421	0.510	3.946
SNV 2 4 4SG	0.892	1.861	0.540	3.803
2 4 4 SG SNV	0.730	2.860	0.524	3.321

N1N2N3: Derivative order, Number of left smoothing points, Number of right smoothing points; SG: Savitzky Golay Derivative; SNV: Standard Normal Variate; R^2: Coefficient of determination of Calibration; SEC: Standard Error of Calibration.

Table 4 summarizes the calibration statistics for CP. Most math treatments reach R^2 values lower than 0.5 for raw alfalfa samples. This could be related to the heterogeneity of alfalfa forage, with two clearly different parts, the leaf and the stem. The leaf is part of the plant containing protein fraction. However, it is important to remark that using the reduced range, the spectra math pretreatment of SNV for scatter correction and the second Savitzky Golay derivative (the same math pretreatment as for NDF), R^2 values of 0.885, with a SEC of 0.377 were achieved. A typical SEL for reference CP analysis is around 0.210 [22].

Table 4. Calibration statistics of NIR multivariate models for Crude Protein quantification

Raw alfalfa				
Wavelength Range:	**901-1600 nm**		**901-1700 nm**	
Math pretreatment	**R^2**	**SEC**	**R^2**	**SEC**
1 4 4 SG	0.742	0.510	0.884	0.428
2 4 4 SG	0.262	0.911	0.608	1.262
SNV 1 4 4SG	0.307	1.314	0.156	1.524
1 4 4 SG SNV	0.678	0.842	0.257	1.378
SNV 2 4 4SG	0.885	0.377	0.345	0.855
2 4 4 SG SNV	0.328	0.812	0.318	1.368
Grounded Alfalfa				
Wavelength Range:	**901-1600 nm**		**901-1700 nm**	
Math pretreatment	**R^2**	**SEC**	**R^2**	**SEC**
1 4 4 SG	0.671	0.986	0.706	0.927
2 4 4 SG	0.906	0.530	0.290	1.014
SNV 1 4 4SG	0.773	0.816	0.790	0.650
1 4 4 SG SNV	0.734	0.882	0.723	0.651
SNV 2 4 4SG	0.862	0.660	0.216	1.145
2 4 4 SG SNV	0.820	0.746	0.179	1.433

N1N2N3: Derivative order, Number of left smoothing points, Number of right smoothing points; SG: Savitzky Golay Derivative; SNV: Standard Normal Variate; R^2: Coefficient of determination of Calibration; SEC: Standard Error of Calibration.

Developed models carried out with ground samples and using a reduced range (901-1600) showed statistics around 0.7 or higher, with SEC values between 0.530 and 0.986. Considering these results, it is worth mentioning that, even though the homogeneity of ground samples gives better calibration statistics, NIRscan nano reached acceptable values when scanning raw samples.

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Feeding animals with minerals is a common practice, however, an abnormal mineral content (MC) there, is a big probability of contamination with soil, which is not desirable for animal feeding systems. To quantify MC in alfalfa forages, 24 different calibration models have been developed assaying different math pretreatments of spectra data. Statistics of proposed PLS models are shown in Table 5. As stated before, the reduced range gave better calibration statistics than the full one. Comparing math pretreatments, scatter correction applied after the derivatization procedure increased R^2 values and reduced SEC. The highest R^2 and the lowest SEC values were 0.861/0.219 and 0.867/0.318 for raw and ground samples respectively.

Table 5. Calibration statistics of Mineral Content multivariate models

Raw alfalfa				
Wavelength Range:	**901-1600 nm**		**901-1700 nm**	
Math pretreatment	**R^2**	**SEC**	**R^2**	**SEC**
1 4 4 SG	0.524	0.503	0.211	0.572
2 4 4 SG	0.619	0.492	0.129	0.861
SNV 1 4 4SG	0.783	0.374	0.734	0.464
1 4 4 SG SNV	0.502	0.579	0.312	0.491
SNV 2 4 4SG	0.675	0.409	0.679	0.434
2 4 4 SG SNV	0.861	0.219	0.687	0.444
Grounded Alfalfa				
Wavelength Range:	**901-1600 nm**		**901-1700 nm**	
Math pretreatment	**R^2**	**SEC**	**R^2**	**SEC**
1 4 4 SG	0.650	0.530	0.652	0.506
2 4 4 SG	0.770	0.435	0.243	0.819
SNV 1 4 4SG	0.570	0.586	0.670	0.519
1 4 4 SG SNV	0.867	0.318	0.347	0.723
SNV 2 4 4SG	0.604	0.566	0.591	0.579
2 4 4 SG SNV	0.781	0.424	0.301	0.625

N1N2N3: Derivative order, Number of left smoothing points, Number of right smoothing points; SG: Savitzky Golay Derivative; SNV: Standard Normal Variate; R^2: Coefficient of determination of Calibration; SEC: Standard Error of Calibration.

These NDF, CP and MC calibration model statistics, obtained with the NIRscan nano prototype, are similar to those acquired with commercial portable instruments using a wavelength range similar to this evaluated in this work [22,23]. SEC values are in accordance with laboratory results [22] and the effect of the sampling procedure has been studied comparatively in this work. As a summary of the obtained results, Table 6 selects the best models obtained for each sampling procedure (raw or ground alfalfa) and parameter. As can be seen, the second derivative is the best of the assayed pretreatments that provide satisfactory results for nutritive value quantification.

Table 6. Statistical analysis of alfalfa nutritive values (N = 57).

Parameter	Sampling	Math pretreatment	Range (nm)	R^2	SEC
NDF	Raw	SNV 2 4 4 SG	900-1600	0.955	1.066
	Ground	SNV 2 4 4 SG	900-1600	0.892	1.861
CP	Raw	SNV 2 4 4 SG	900-1600	0.885	0.377
	Ground	2 4 4 SG	900-1600	0.906	0.530
MC	Raw	2 4 4 SG SNV	900-1600	0.861	0.219
	Ground	1 4 4 SG SNV	900-1600	0.867	0.318

N1N2N3: Derivative order, Number of left smoothing points, Number of right smoothing points; SG: Savitzky Golay Derivative; SNV: Standard Normal Variate; R^2: coefficient of determination for calibration, SEC: standard error of calibration, r^2: coefficient of determination for cross-validation,

SECV: standard error of cross-validation, NDF: Neutral detergent fibre; MC: Mineral content; CP: crude protein.

5. Conclusions and Future Work

This work reports on a miniaturized optoelectronic measurement system for decentralized agrifood quality control. Heterogeneous forage (alfalfa) has been selected as a model to evaluate the precision of instrumental measures (spectra collected) and the effect of sampling presentation (raw or ground) on calibration statistics. Results have revealed that homogeneous forage samples (those milled) allow reaching better calibration models than those scanned in their raw form (heterogeneous). However, for all sampling procedures, it has been possible to obtain satisfactory calibration to quantify nutritive parameters.

This technological proposal combined with proper chemometric tools offers an excellent alternative to on-site, real-time, and non-destructive analysis of agrifood products. In addition to providing owners and technicians with easy-to-use, the proposed measurement system offers a means of evaluating forage quality, taking more samples without incurring expenses, obtaining real-time results, and making quick decisions.

In the future, Internet access will be useful for sharing information between different NIRscan nano instruments. These instruments can be standardized and share calibration models to quantify nutritive parameters. Moreover, a common database of spectra collected with different instruments can be used as a reference to increase sample variability and improve calibration statistics.

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